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NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA

GB 31604.21-2016

**National food safety standard –
Food contact materials and articles –
Determination of migration of terephthalic acid**

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Foreword

This Standard replaces GB/T 23296.10-2009, *Food contact materials - Polymer - Determination of terephthalic acid in food simulants - High performance liquid chromatography*, and SN/T 2184-2008, *Food contact materials - Polymer materials - Determination of terephthalic acid in food simulants - HPLC*.

Compared with GB/T 23296.10-2009 and SN/T 2184-2008, the major changes of this Standard are as follows:

- the standard name is changed into “National food safety standard - Food contact materials and articles - Determination of migration of terephthalic acid”;
- the preparation of food simulant test solution is changed;
- the expression of analysis results is changed;
- the detection limits and quantitation limits are changed.

National food safety standard –

Food contact materials and articles –

Determination of migration of terephthalic acid

1 Scope

This Standard specifies the method for the determination of migration of terephthalic acid in food contact materials and articles.

This Standard applies to the determination of migration of terephthalic acid in food contact materials and articles.

2 Principle

Terephthalic acid in food simulants is separated using high performance liquid chromatograph and then tested using ultraviolet detector. The food simulants of water-based, acidic and alcoholic types are sampled and tested directly; and the food simulants of olive oil medium is extracted using diluted hydrochloric acid solution before being sampled and tested. Phthalic acid is used as the internal standard and the internal standard method is used for quantitation.

3 Reagents and materials

Unless indicated otherwise, all reagents used for this method are analytically pure and the water is grade one water specified in GB/T 6682.

3.1 Reagents

3.1.1 Sodium acetate trihydrate ($C_2H_3O_2Na \cdot 3H_2O$).

3.1.2 Sodium bicarbonate ($NaHCO_3$).

3.1.3 Normal heptane ($n-C_7H_{16}$).

3.1.4 Methyl alcohol (CH_3OH): chromatographically pure.

3.1.5 Isopropyl alcohol (C_3H_8O).

3.1.6 Phosphoric acid (H_3PO_4).

water-based, acidic and alcoholic food simulants are respectively 0.0 mg/L, 0.8 mg/L, 1.6 mg/L, 4.0 mg/L, 8.0 mg/L and 16.0 mg/L; and the internal standard concentrations are all 8.0 mg/L.

3.4.4 Olive oil medium standard working solution: weigh respectively 50 g of olive oil to pour in six 250 mL separating funnels; accurately transfer 2.0 mL of terephthalic acid standard intermediate solution in; add 50 mL of normal heptane after mixing up; and then mix up. Then add 20 mL of sodium bicarbonate solution; fully shake up for 1 min; and allow to stand for layering. Use a 100 mL beaker to collect the lower layer water phase; then add 20 mL of sodium bicarbonate solution to extract the upper layer oil phase once again. Allow to stand after shaking up, use a beaker to collect the lower layer water phase after two phases separates; and combine the two water-phase extraction solutions. Use an injection syringe or vacuum pump (flow velocity 10 mL/min~20 mL/min) to make the extraction solution pass through the solid-phase extraction C₁₈ column. Collect the filtrate in a 50 mL volumetric flask; add 1.0 mL of 50% acetic acid solution; and use water to add to scale. Take 1 mL~2 mL of the filtrate to filter using a filter membrane. The concentrations of the standard working solutions of terephthalic acid are respectively 0 mg/kg, 0.8 mg/kg, 1.6 mg/kg, 4.0 mg/kg, 8.0 mg/kg and 16.0 mg/kg; and the internal standard concentrations are all 8.0 mg/kg.

3.4.5 Phthalic acid internal standard stock solution (1 000 mg/L): accurately weigh 0.1 g (accurate to 0.1 mg) of phthalic acid standard substance; use 10 mL of isopropyl alcohol to dissolve; and then transfer the solution to a 100 mL volumetric flask and add isopropyl alcohol to scale. The period of validity is 6 months.

3.4.6 Terephthalic acid internal standard intermediate solution (200 mg/L): take 5 mL of phthalic acid internal standard stock solution to pour into a 25 mL volumetric flask; and use methyl alcohol to add to scale.

4 Apparatus

4.1 High performance liquid chromatograph: equipped with ultraviolet detector and 10 µL quantitative loops.

4.2 Analytical balance: of sensitivity 0.1 mg and 0.1 g.

4.3 pH meter.

4.4 Solid-phase extraction C₁₈ column: octadecyl silane (ODS) 400 mg.

4.5 Hydrophilic microporous filter membrane: 0.45 µm.

5 Analytical procedures

5.1 Sample migration test

e) sample volume: 10 μ L;

f) ultraviolet detector: of wavelength 242 nm.

5.4.2 Plotting of standard working curves

Carry out determination of standard working solutions in turn in accordance with the determination conditions listed in 5.4.1. Plot respectively standard working curves using the terephthalic acid concentrations in standard working solutions as the abscissa, in mg/L or mg/kg, and the peak area ratio of terephthalic acid/phthalic acid as the ordinate. For the standard solution chromatograms refer to Annex A.

5.4.3 Test solution determination

Inject blank solutions and food simulant test solutions in turn, deducting blank values; and obtain the peak area ratio of terephthalic acid/phthalic acid.

6 Expression of analysis results

Obtain the terephthalic acid concentration of test solution from standard curves; and calculate migration in accordance with GB 5009.156; and then obtain the migration of terephthalic acid in food contact materials and articles. The calculation results are rounded off to two significant digits.

7 Confirmation

When the above-mentioned results exceed specific migration limits, use the liquid chromatography-tandem mass spectrometry or other effective methods for confirmation. For the reference conditions for the liquid chromatography-tandem mass spectrometry see Annex B and Annex C.

8 Precision

The absolute difference obtained from two independent determination results under repeatable conditions shall not be 10% exceeding the arithmetic mean value.

9 Others

The detection limit for terephthalic acid in water-based, acidic and alcoholic simulants is 0.3 mg/L; and the detection limit for terephthalic acid in olive oil simulants is 0.3 mg/kg.

The quantitation limit for terephthalic acid in water-based, acidic and alcoholic simulants is 1.0 mg/L; and the quantitation limit for terephthalic acid in olive oil simulant